Inductively-Coupled-Plasma Mass Spectrometry

Inductively-coupled plasma mass spectrometry (ICP-MS) provides quantitative determinations of elements at concentrations ranging from major constituent to parts-per-trillion. It is particularly useful as a trace analysis technique.

ICP-MS is an excellent method for identifying elements and elemental impurities in solution. Approximately 70 elements can be determined by this scanning technique. The isotopic composition of elemental constituents can also be determined.

Qualitative Analysis. The presence of an element is established by the presence of isotopes of a specific mass-to-charge ratio. In most cases, unequivocal elemental assignment is possible because of the presence of several isotopes at specific masses with known isotopic ratios.

Quantitative Analysis. The concentration of an element is generally measured by reference to a set of standard solutions of known concentration. Using an internal standard technique, measurement with a relative precision and accuracy of 1% is typical.

Isotopic Measurements. Isotopic ratios, typically accurate to 0.1–0.5%, may be measured using ICP-MS. This requires characterization of mass discrimination effects within the spectrometer for the particular element in question, using materials of known isotopic composition. Additional correction for detector system deadtime is also generally required.

Principle of Technique

Samples are introduced, generally as a nebulized solution, into an argon-supported, rf plasma discharge. The energetic conditions associated with the high temperature plasma are sufficient to evaporate and atomize both solvent and solute. A fraction of these atomic species undergo ionization within the discharge. A portion of the plasma is then extracted into a quadrupole mass filter through a pair of sampling orifices. Several stages of differential pumping establish the required vacuum gradient between the ambient pressure used for operation of the plasma discharge and the high vacuum conditions required for mass filter operation. Within the vacuum system, ions are focused into a beam by a set of ion lenses, separated on the basis of mass-to-charge ratio by means of a quadrupole mass filter, and detected by an electron multiplier operating in a pulse-counting mode.

Samples

Form. Liquids, usually aqueous solutions, can be analyzed as received or after dilution.

Size. 5 mL of solution is required for routine analysis. The minimum sample size that must be present varies by element but must be sufficient to yield 500–10,000 pg of analyte.

Preparation. Solids must be dissolved in a suitable medium usually an acidic, aqueous solution.

Limitations

Because of its high sensitivity, major constituent analysis requires several stages of serial dilution. Solid materials must be dissolved in an aqueous
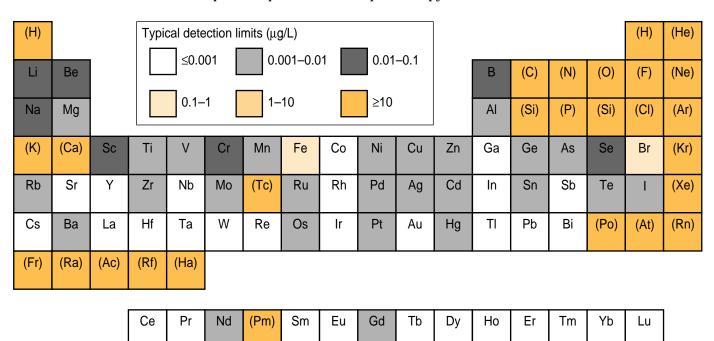
Examples of Applications

- Quantitative analysis of lanthanide dopants in optical materials.

- Qualitative identification and quantitative determination of trace impurities in optical materials.

- Determination of naturally occurring metals in soil and water.

- Isotopic and impurity measurements in AVLIS-enriched materials (e.g., uranium).

- Trace level determinations of metals in biological fluids.

solvent. No direct chemical speciation or structural information is obtained. Coincidental isotopic overlap in the mass spectrum can limit the applicability of this technique. However, this is seldom a problem for elements with isotopic masses greater than 80 daltons.

Estimated Analysis Time

Including instrument startup time, analysis of a single-solution sample generally requires 2 h. Quantitative determinations usually require 3 to 4 h for preparation of samples and standards. Qualitative analysis and data interpretation varies from 30 min to several days, depending on sample matrix and mass spectral complexity.

Capabilities of Related Techniques

Inductively-coupled-plasma emission spectroscopy (ICP-AES) and DC-plasma optical emission spectroscopy (DCP-OES) are comparable multielement techniques but they are generally less sensitive than ICP-MS. For certain elements (e.g., transition elements, alkali, and alkaline earths), ICP-AES and DCP-OES may be more suitable because of mass spectral interferences. Atomic absorption spectrometry (AAS) may be applicable to some elements that suffer specific interferences by ICP-MS. However, AAS is inherently a single-element technique.

Solution chemistry techniques are capable of higher accuracy and precision, and may be applicable to certain samples where macroscopic quantities are available. Dc-arc optical emission spectroscopy and x-ray fluorescence spectrometry may be applicable but are less sensitive than ICP-MS. They can be used to analyze solids directly without dissolution.

ICP-MS is typically used to determine the elements above except those in parenthesis (because of interferences, lack of available standards, or other factors). Shaded blocks indicate reporting limits (μg/L) for careful quantitative work under optimum conditions.